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TECHNICAL REPORT ARAED-TR-88018

HAZARD CLASSIFICATION OF LIQUID PROPELLANTS

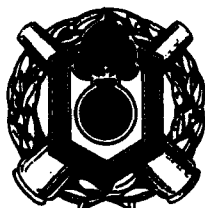
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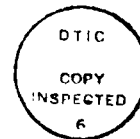
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CONTENTS

	Page
Introduction	1
Literature Search and Surveys	2
Methodology	3
Screening Tests	3
Conduct Screening Tests	5
Conclusions and Recommendations	7
References	21
Distribution List	23

Figures

1	Hazard classification methodology	13
2	Explosive reaction methodology	13
3	No-go reaction methodology	13
4	Card cap test configuration	14
5	Liquid sample holder	14
6	Thermal stability bomb assembly	14



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INTRODUCTION

The hazards classification of solid propellants and explosives was conducted in accordance with the requirements stipulated in accepted manuals such as the DOD Manual TB 700-2. The classification of liquid propellants, however, poses a major problem due to the lack of a formal test procedure or protocol for liquid propellants. The safety community has an interim classification of liquid propellants as Class B. Being designated a liquid propellant, it was automatically classified as an explosive. As such, the DOD Manual TB 700-2 was used in establishing this classification. The manual explicitly states that it applies only to ammunition and explosives other than liquids. Therefore, the hazard classification of these liquid propellants is based on results of tests that were designed strictly for solid propellants.

It is critical that any tests designed for liquid propellants be based on the size of the actual system, the confinement encountered, and the potential modes of ignition that can exist. Many of the tests used in the past for evaluating liquid propellants dealt with quantities too small in size to accurately evaluate the sensitivity of a material to an external stimuli. Liquids, under shock loading generate hydraulic pressures, and these pressures are contingent upon the critical mass, the degree of confinement, and the density of the material. Too often, tests were selected in the evaluation of materials that did not simulate the modes of ignition that were encountered in manufacture, transportation, storage, and field use. As a remedy for this problem, this program was conducted to develop a hazards classification protocol for liquid propellants used in gun applications.

The objective of this program was to evaluate presently used or available test methods and to develop test procedures for the establishment of the sensitivity of liquids. In accomplishing this objective, Southwest Research Institute (SwRI) conducted the following specific tasks:

- (1) SwRI conducted a literature survey to establish the different modes of ignition that could occur during the various stages of manufacture, transportation, operational use, and storage of HAN-based propellants. As part of this task, SwRI interfaced with safety community personnel from the Department of Defense Explosives Safety Board, Alexandria, VA; the Department of Transportation, Washington, DC; the Ballistics Research Laboratory, Aberdeen, MD; the Army Research, Development and Engineering Center, Picatinny Arsenal, NJ; and the AMC Field Safety Activity, Charleston, IN, to discuss and obtain available data on the hazards classification of liquid propellants.

- (2) SwRI developed a methodology for establishing a hazards classification procedure.

(3) A series of screening tests were selected and recommended for possible use in the classification of liquid propellants.

(4) A series of screening tests were conducted on liquid propellants, LGP 1846.

Literature Search and Surveys

To develop a set of viable test procedures for liquid propellants, SwRI conducted an in-depth literature search and review and also attended several meetings with the safety community personnel. A number of reports identifying test procedures were evaluated (ref 1 through 5) and a table listing these key test procedures was developed (table 1). Test procedures are identified for both the solid and the liquid propellants. In addition to the review, SwRI evaluated the NATO AOP-7 706 manual (ref 6) to evaluate if the characteristics and qualification tests identified in the manual were applicable for the hazards classification of liquid propellants.

NATO AOP-7 cites a scheme for the classification of liquids, qualification tests for determining the hazards classification of liquid propellants and the characterization requirements essential for each liquid propellant for acceptance (ref 6). The qualification tests for the hazard classification of liquid propellants are:

- Unconfined burning (Bonfire tests)
- Impact tests
- Card gap test
- Minimum pressure for vapor phase ignition
- Flash point test
- Adiabatic compression
- Detonation velocity

All of these tests with the exception of minimum pressure for vapor phase ignition test and the flash point test were considered viable tests for qualifying the present liquid propellants. These two tests were eliminated from consideration in establishing a hazards classification of the hydroxyammonium nitrate (HAN) based liquid propellants because the carrier system is water. Where volatile organic solvents are used, these tests should be considered.

The tests identified in table 1 and those listed above were evaluated for applicability and the following series of qualification tests were selected:

- Card gap test
- Impact test
- Thermal stability test
- Electrostatic test

Methodology

The use of full-scale tests are normally preferred over laboratory-scale tests; however, full-scale tests can be costly. To make an assessment of the explosive potential of a liquid propellant, small-scale laboratory tests can provide reliable information if the environmental modes of ignition are addressed. The modes of ignition considered critical for evaluating liquid propellants on a laboratory scale are thermal, compression ignition, impact, and electrostatic. Critical mass and diameter are parameters that must be obtained by the use of full-scale test methods. As shown in figure 1, once a liquid propellant demonstrates a positive explosive reaction in one of the laboratory screening tests, further testing will be continued to determine if the material is a Class A or B explosive (fig. 2). If the liquid propellant does not produce explosive reactions in all the screening tests (fig. 3), then the materials are subjected to the full-scale critical mass and diameter tests.

Screening Tests

A series of screening tests were selected for the classification of liquid propellants. These screening tests include: card gap test, impact test, thermal stability test, and electrostatic test.

Card Gap Test

The card gap test is the standard DOD TB 700-2 test designed to determine the sensitivity of a material to the shock from a detonation. A typical card gap tester is shown in figure 4. The test apparatus consists of a one-piece 4.75-cm outside diameter by 14-cm long (1.875 in. O.D. by 5.5 in. long) mild steel tube, two pentolite pellets (each weighing approximately 60 grams), a J-2 blasting cap, a 15 by 15 by 1 cm (6 in. by 6 in. by 0.375 in.) mild steel witness plate, cellulose acetate (or equivalent) cards 5 cm in diameter by 0.025 cm thick (2 in. diameter by 0.01 in. thick). Four small pieces of plastic 0.16 cm by 1.27 cm (0.0625 in. by 0.5 in.) are used to support the tube and to maintain a 0.158 cm (0.0625 in.) air gap between the test sample and the witness plate. The arrangement of the components is similar to that shown in figure 4 with the exception that the cellulose cards and the cardboard tube are omitted in the first test. Detonation is indicated when a clean hole is cut in the witness plate. Should no detonation occur in the first test, it will be repeated two times for a total of three tests. If detonation does occur, then a series of tests using the acetate cards will be conducted. The acetate cards are placed between the liquid propellant tube and the pentolite boosters (fig. 4). The first test is performed using eight cards, and if a detonation occurs, then the number of cards is doubled for the second test. If not detonation occurs, then the number of cards is halved. Doubling the number of cards will be continued until no detonation occurs. When the number of cards is reached that prevents detonation, the next test is performed with the number of cards reduced by half the preceding increment of increased (i.e., if detonation occurs at 32 cards but not at 64 cards, then the next test

is run with 48 cards). If detonation occurs at the reduced number of cards (48 cards in the example given) then the number of cards will be increased by one-half the preceding increment (i.e., from 48 cards to 56 cards). This test procedure will be continued until the point of 50% probability of detonation is obtained. Normally, a maximum of 12 tests is required to determine the 50% value.

Detonation is indicated when a clean hole is cut in the witness plate. The measure of charge sensitivity is the length of the attenuation (gap length) at which there is a 50% probability that detonation will occur as detailed in the above procedure.

Impact Test

The drop weight test is the standard JANNAF test designed primarily to determine the sensitivity of a material to impact. A typical impact test apparatus is shown in figure 5. The liquid test sample (0.03 ml) is enclosed in a cavity formed by a steel cup, an elastic ring, and a steel diaphragm. A piston rests on the diaphragm and carries a vent hole which is blocked by the steel diaphragm. A 2-kg weight is dropped onto the piston. A positive result is indicated by puncture of the steel diaphragm accompanied by a loud report or by severe deformation of the diaphragm and evidence that the sample was completely consumed. Data are reported as the height which yields a 50% probability of initiation.

JANNAF Thermal Stability Test

The JANNAF thermal stability study is the standard test designed by the ICRPG for testing the thermal sensitivity of propellants. The test fixture is a stainless-steel cylinder 0.56 cm in diameter by 3.81 cm long (0.22 in. diameter by 1.5 in. long) closed at the bottom with a shielded thermocouple and a compression fitting (fig. 6). The fixture is charged with 0.5 cm³ of sample and closed at the top with a stainless-steel diaphragm, 7.6 mm (0.003 in.) thick. The assembly is then placed in a bath which is heated at a constant rate of 10°C/min. A second thermocouple and an X-Y recorder are connected with the sample thermocouple so as to yield a plot of differential temperature (sample temperature minus bath temperature) versus bath temperature. Exothermic reactions appear as positive peaks; endothermic reactions appear as negative peaks. Results are reported in terms of the temperatures at which significant thermal activity is observed, and the temperature at which the burst disk yields.

Electrostatic Test

Electrostatic energy stored in a charged capacitor is discharged to the sample material being tested to determine whether an electrostatic discharge will cause the sample to decompose, flash, burn, etc. The sample is placed on a special holder which assures that the discharge will pass through the sample. The capacitor is charged with a 5000 volt potential; the discharge needle is lowered until a spark is drawn through the

sample (the sample being about 20 mg in size). The standard test interval ranges from 0.0001 microfarads (μf) and 0.00125 J (at 5kV) to 1 μf and 12.5 J (at 5 kV). The test is begun at the 12.5 J (1 μf) level. If results are negative, testing is continued until 20 consecutive negatives are reported at that level. If the result is positive, such as a flash, spark, burn, odor, or noise other than instrument noise, then the next lower test interval is tried until 20 consecutive negative results are reported. The test is normally conducted at a voltage of 5 kV DC or less. An ambient temperature of 18 to 32°C (65 to 90°F) and relative humidity not exceeding 40% are maintained.

Conduct Screening Tests

Card Gap Tests

A series of tests were conducted by SwRI on LGP 1846. The results of these tests are presented in table 2. Four tests were conducted using the LGP 1846 (one test was conducted with a longer mild steel tube having an L/D of 3 times the standard mild steel tube, i.e., 42 cm). A series of tests were conducted on LGP 1845 by Hazards Research Corporation in 1981 and have been included for comparison purposes with the result of the tests on the LGP 1846.

The test by SwRI on the LGP 1846 were conducted in accordance with the test procedure described earlier with two exceptions: (1) a polyethylene liner was used to isolate the LGP from the mild steel test tube, thereby precluding any contamination of the LGP, (2) the pentolite disks each weighed approximately 90 grams instead of 60 grams. The test tube with the polyethylene liner prior to being filled with the LGP is shown in figure 7, the liner full of liquid propellant in the mild steel tube in figure 8. On the tests conducted on the LGP 1845, there were no detonations since the witness plates were dished but not punched with a hole. Similarly, the tests involving the LGP 1846 resulted in the witness plates being dished but no holes being cut into the plates. The witness plates for the three tests conducted with the standard mild steel tube are shown in figures 9 through 10. These plates are severely deformed, but no holes have been punched. A test to confirm that the liquid propellant was becoming involved by the detonation of the pentolite and that the damage to the plate was not strictly due to the pentolite charges was conducted. In order to determine what contribution the LP was making, the confirmation was conducted with water instead of liquid propellant. The results of this test (fig. 12) show that the damage to the plate is much less without the LGP. The test conducted with the longer mild steel tube (fig. 13) shows that the witness plate is even less damaged than the plates in the previous tests with the smaller tube, indicating that the detonation is not being propagated through the liquid propellant but instead, is being attenuated.

The tests conducted by Hazards Research on the LGP 1845, however, were conducted using a smaller 10 cm by 10 cm (4 in. by 4 in.) witness plate and did result in reactions at the lower levels. Tests were conducted using larger quantities of attenuation cards up to a maximum of 70 cards with no reaction occurring at the 70-card level. Tests were conducted using the larger 15 cm by 15 cm (6 in. by 6 in.) witness plate and with 70 cards with no reaction. According to the DOD TB 700-2 classification scheme, the criteria for distinguishing between military class 7 (DOT class A) and military class 2 (DOT class B) is the sensitivity value of 70 cards. If the card value for the propellant is greater than 70 cards, then the classification is military class 7. If the card value is less than 70 cards or there is no reaction at 0 cards, then the classification is considered military class 2. According to this criteria, the LGP 1845 and the LGP 1846 are classed as explosives military class 2. This classification protocol does not allow for an accurate hazard assessment for a material when no reaction occurs at zero cards as is the case with the LGP 1846. A material should exhibit an explosive reaction before it is classified as an explosive. To follow the procedure of classification as outlined in the TB 700-2 is inconsistent with the concept of an explosive material.

Impact Tests

A series of impact tests were conducted on LGP 1845 and 1846. The results of these tests are presented in table 3. The drop height value for LGP 1845 and LGP 1846 that resulted in a 50% probability of initiation was 76 cm (30 in.). The impact value reported for nitromethane is 50 cm (20 in.). According to ICC regulations, nitromethane is shipped as a flammable liquid. By comparison, the liquid propellants cited are less sensitive to impact than nitromethane yet are classed as a class B explosive.

Thermal Stability

The thermal stability, under confinement, demonstrates the temperature at which significant thermal activity is observed and the temperature at which a rupture disk yields. The results of the LGP materials (table 4) indicate that the LGP materials produce high gas pressures under conditions of confinement. This is consistent with the normal functions of a gun propellant.

Electrostatic Tests

A series of electrostatic tests were conducted by SwRI in accordance with the test protocol outlined earlier. The test apparatus used for the tests is shown in figure 14; and the sample holder which consists of a brass metal 0.001-in. brass shim stock which was bonded to a 0.16 cm (0.0625 in.) thick high voltage phenolic dielectric material in figure 15. The brass metal was connected to the ground lead of the spark generator. A 0.64 cm (0.25 in.) hole was cut into the insulator exposing the metal plate. The sample liquid material was then placed in the small cylindrical cavity created in the

insulator. The upper electrode was then lowered near the bottom plate (the sample holder), and the charge was then allowed to arc through the liquid sample. Tests were conducted at energy levels of 180 mJ with no reaction. Twenty repeat tests were conducted with no reactions.

CONCLUSIONS AND RECOMMENDATIONS

Based on the results of the literature search and the screening tests performed on the LGP 1846, a number of conclusions and recommendations have been formulated.

According to the hazards classification stipulated in TB 700-2 for the card gap test procedure, if the card value necessary to prevent detonation is less than 70 cards or if there is no reaction at 0 cards, then the classification is considered military class 2. This classification protocol does not allow for an accurate hazard assessment for a material when no reaction occurs at zero cards as was the case with the LGP 1846 tests conducted at SwRI. It is recommended that a material be classified as an explosive only after it has exhibited an explosive reaction during the classification tests. To follow the procedure of classification as outlined in the TB 700-2 is inconsistent with the concept of an explosive material.

Since the earlier card gap tests conducted by Hazards Research Corp. on the LGP 1845 propellant using the larger 15.2 cm by 15.2 cm (6 in. by 6 in.) witness plate were run only with 70 cards, it is recommended that a series of tests be performed using the larger witness plate and with no attenuation cards. If a reaction occurs, then the normal progression of increasing the attenuation cards until no reaction occurs or 70 cards is reached should be performed.

It is recommended that small scale tests be performed on liquid propellants before performing any of the full scale tests. Small scale tests such as the impact test, the electrostatic test, and the thermal stability tests performed by SwRI and by Hazards Research demonstrated the feasibility of the use of small scale tests.

Small scale tests such as the card gap test have been demonstrated effectively on certain liquid propellants such as the LGP 1845 tests performed by Hazards Research Corp. However, these tests may need to be modified for other materials such as the LGP 1846 in order to better determine how the material is responding to an external stimuli. The use of a longer tube than the 14 cm (5.5 in.) tube currently used in the card gap test is one of the modifications that needs to be further explored.

Table 1. Applicable safety testing criteria

Tests	Hazardous liquids	Hazardous solids
Impact tests		
Drop weight (refs 1, 2, 3)	*	*
Adiabatic compression (ref 2)	*	*
Compression-ignition sensitivity (ref 2)	*	
Low amplitude compression (ref 2)	*	
Thermal tests		
Ignition temperature (ref 2)	*	*
Flash point (refs 1, 2)	*	*
Differential thermal analysis (ref 2)	*	
Thermal surge (ref 2)		
Thermal stability (refs 1, 2, 3) (10°C/min)	* ^a	
Thermal stability scan (ref 1) (2°C/min)	*	
Long term thermal stability (ref 1) (100°C for 48 hr)	*	*
Thermal explosion scan (ref 4)	*	*
Autoignition temperature (ref 1)	*	*
Shock energy input tests		
Card gap (refs 1, 2, 3)	*	*
Impedance mirror (ref 2)	*	
Shock confinement tests (ref 2)	*	
Storage hazards tests		
Single package test (ref 3)		*
Stack test (ref 3)		*
External fire stack test (refs 1, 3)	* ^b	*
Blast test (ref 3)		*
Fragment hazard test (ref 3)		*
Thermal effects test		
Thermal radiation field (ref 3)		*
Firebrands (ref 3)		*

Table 1. (cont)

Tests	Hazardous liquids	Hazardous solids
Other		
Friction (refs 4, 5)	*	*
Electrostatic discharge (refs 4, 5)	*	*
Dust explosibility (ref 4)	N/A	*
Detonation test (ref 3)		*
Detonation velocity (ref 1)	*	
Deflagration potential (ref 1)		*
Ignition and unconfined burning (ref 3)		*
Trauzl block (refs 1, 5)	*	*
Cap sensitivity (ref 5)	*	
Bullet Impact (ref 5)	*	*

^aThermal stability tests should also be performed with the sample under pressure to determine the effects of pressure on sensitivity.

^bLimited bonfire and unconfined burning tests have been performed on liquid propellants using 1-gal aluminum cans. Stack test, however, have not been performed.

Table 2. Card gap test

<u>Test no.</u>	<u>Material type</u>	<u>No. of cards</u>	<u>Results</u>
1	LGP 1846	0	Witness plate severely deformed, no hole punched in plate.
2	LGP 1846	0	Witness plate severely deformed, no hole punched in plate.
3	LGP 1846	0	Witness plate severely deformed, no hole punched in plate
4	Water	0	Witness plate slightly deformed.
5	LGP 1846	0	Witness plate slightly deformed, no hole punched in plate.
1	LGP 1845	70	Witness plate dished but not punched with a hole.
2	LGP 1845	70	Witness plate dished but not punched with a hole.

Table 3. Impact tests

<u>Sample identification</u>	<u>Drop height (in.)</u>		
	<u>0%</u>	<u>50%</u>	<u>100%</u>
1. LGP 1845	28	30.0	31
2. LGP 1946	29	30.5	33

Table 4. JANNAF thermal stability

<u>Sample identification</u>	<u>Temperature of major exotherm onset (°C)</u>	<u>Remarks</u>
1. LGP 1845	135	Very sharp and rapid exotherm, burst disk
2. LGP 1846	120	Very sharp and rapid exotherm, burst disk

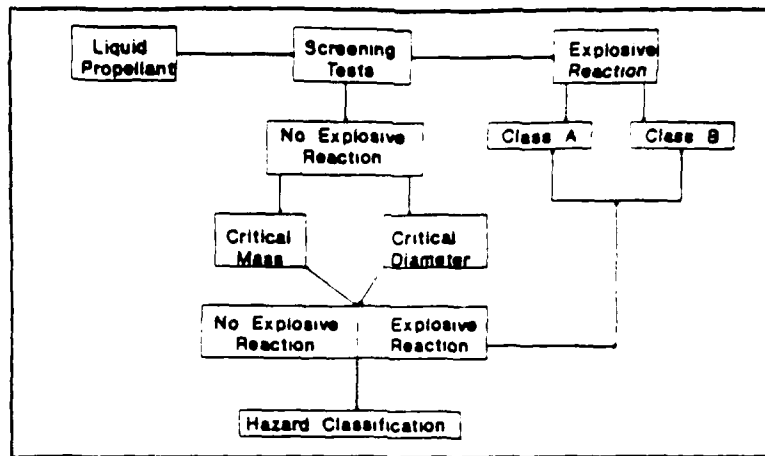


Figure 1. Hazard classification methodology

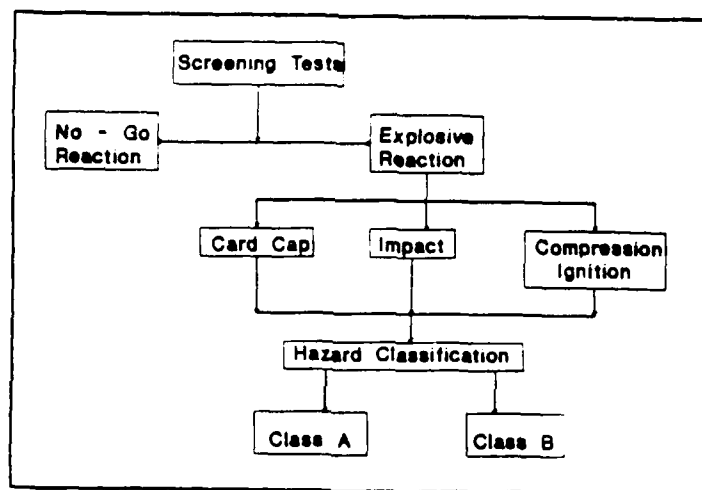


Figure 2. Explosive reaction methodology

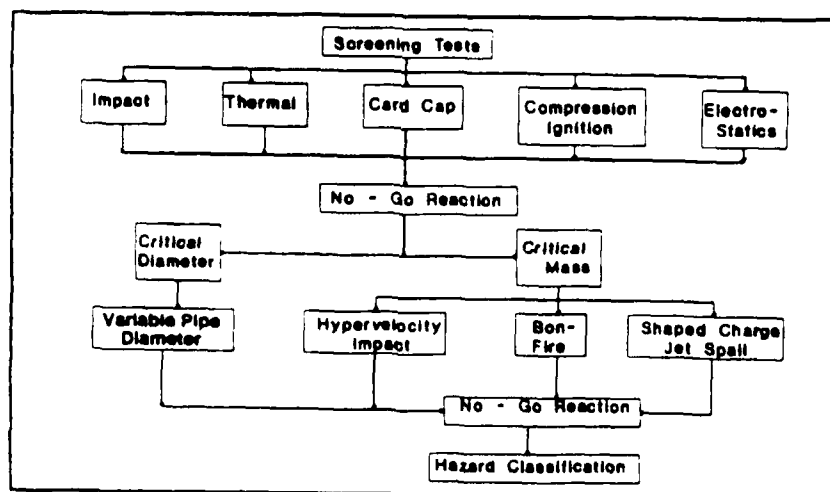


Figure 3. No-go reaction methodology

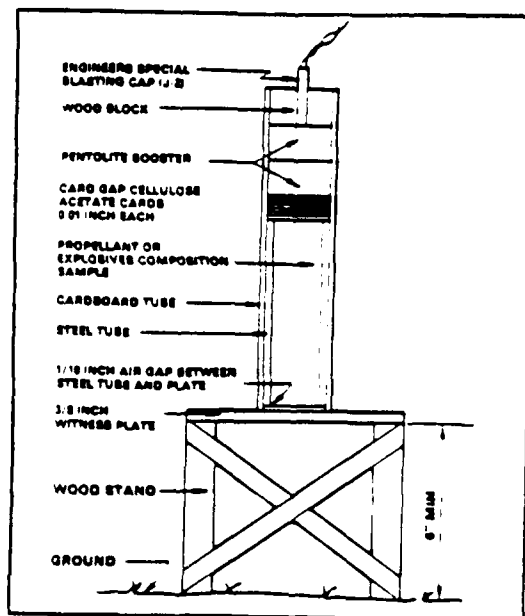


Figure 4. Card cap test configuration

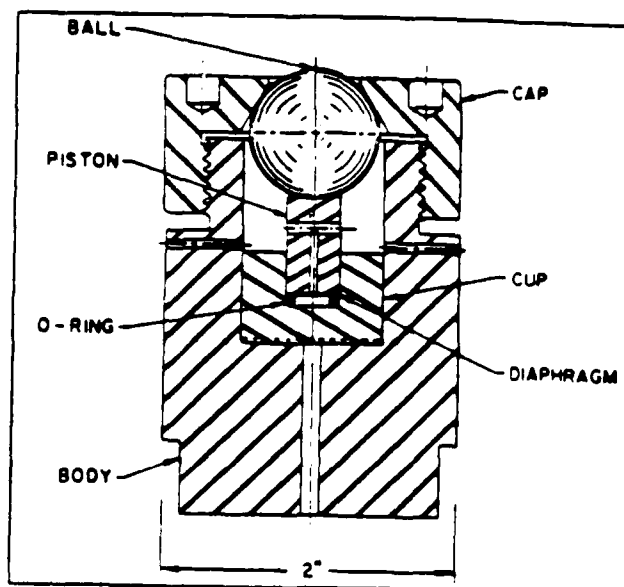


Figure 5. Liquid sample holder

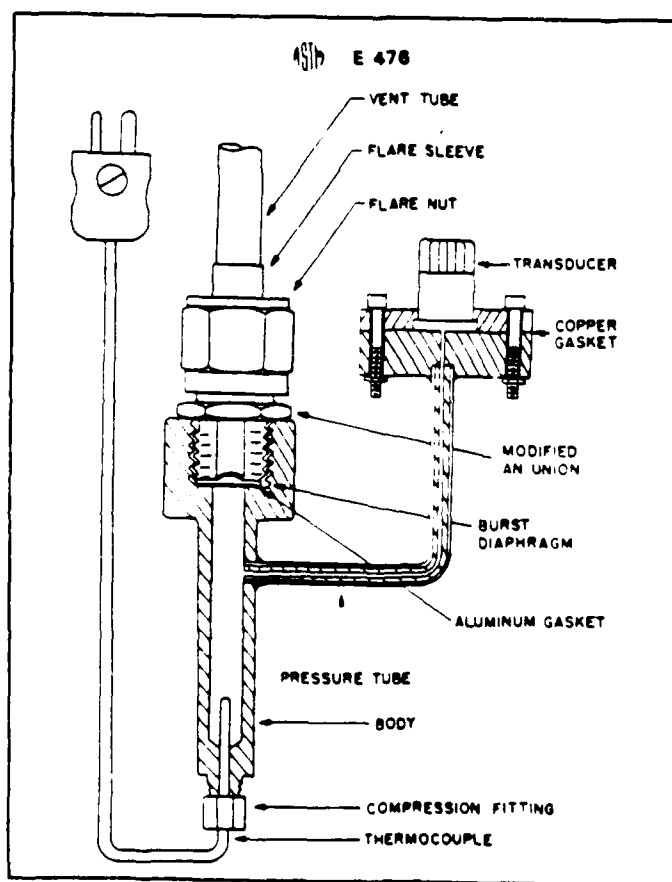


Figure 6. Thermal stability bomb assembly



Figure 7. Mild steel tube and liner



Figure 8. Tube with liner full of LGP 1846

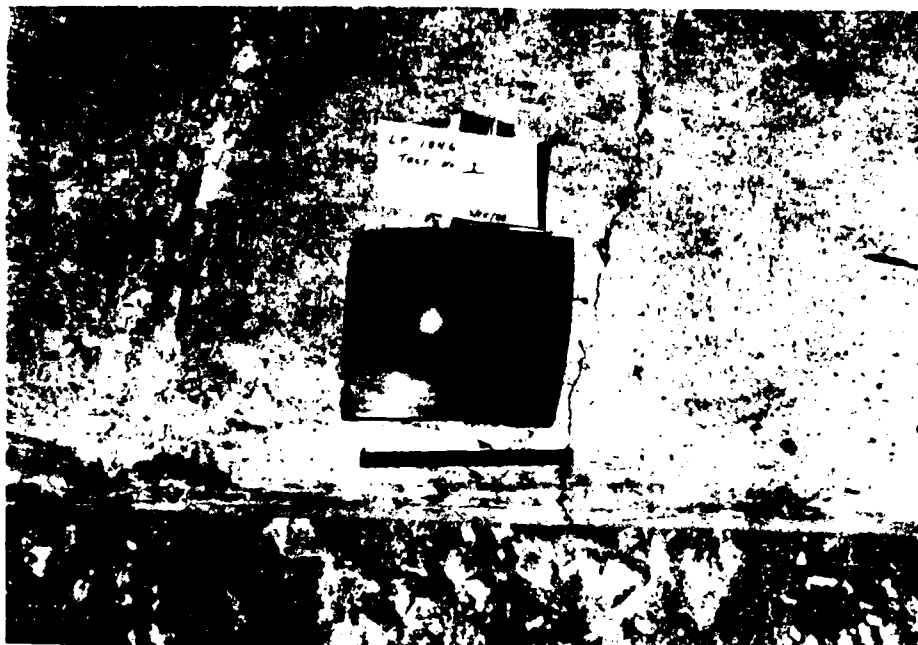


Figure 9. Witness plate, test no. 1



Figure 10. Witness plate, test no. 2



Figure 11. Witness plate, test no. 3

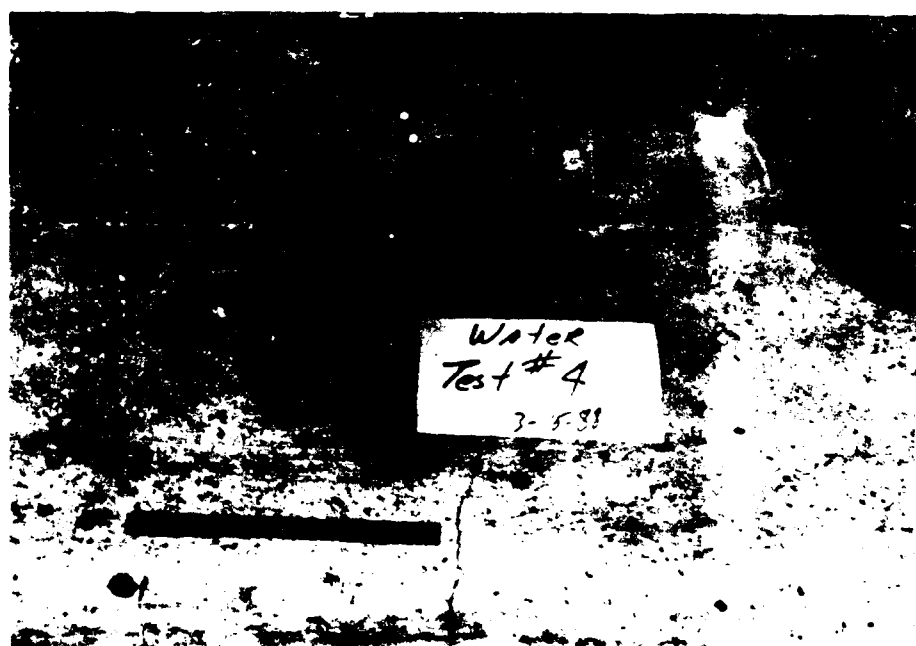


Figure 12. Witness plate, test no. 4 conducted with water instead of LGP 1846



Figure 13. Test conducted with LGP 1846 with longer tube

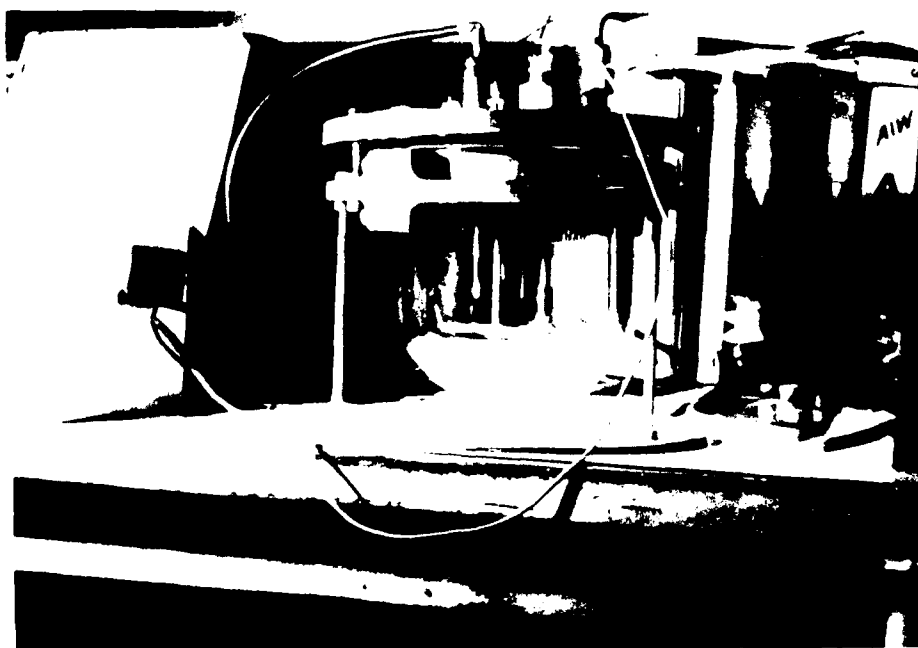


Figure 14. Electrostatic test apparatus

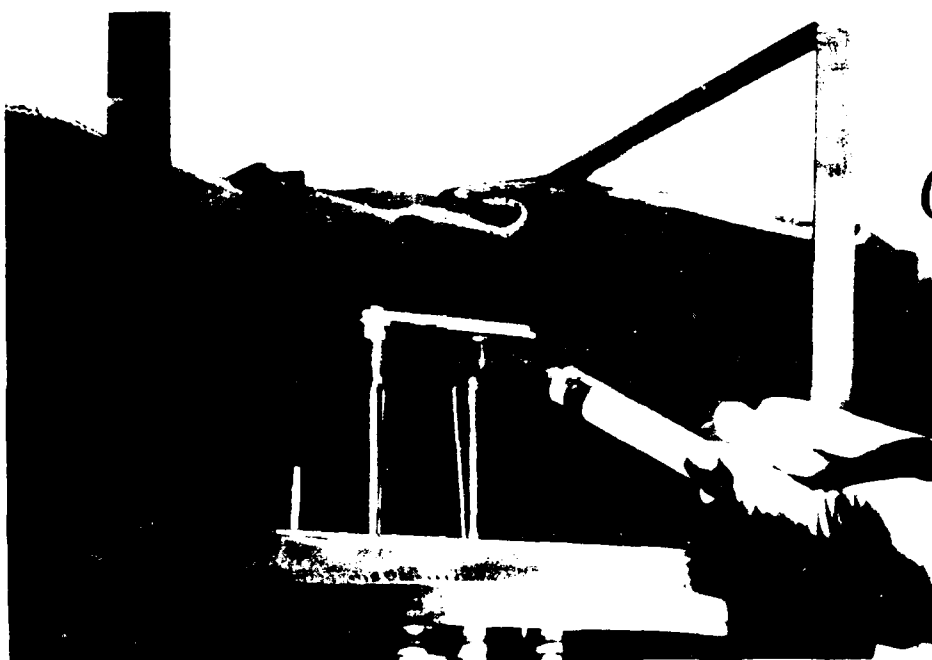


Figure 15. Sample being placed in holder

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